

# **STUDIES IN VISCOSITY OF RAPIDLY SETTLING SUSPENSIONS**

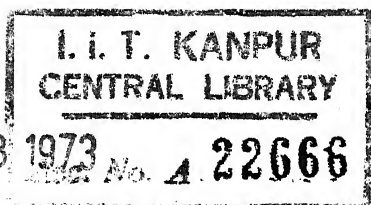
A Thesis Submitted  
In Partial Fulfilment of the Requirements  
for the Degree of  
MASTER OF TECHNOLOGY

00013

BY  
SOM DUTTA KHULLAR

to the

DEPARTMENT OF CHEMICAL ENGINEERING  
INDIAN INSTITUTE OF TECHNOLOGY KANPUR  
DECEMBER, 1972



Thesis

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TO

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ALL OTHERS WHO LOVE ME

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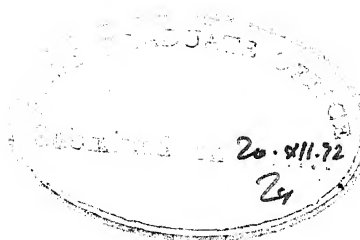
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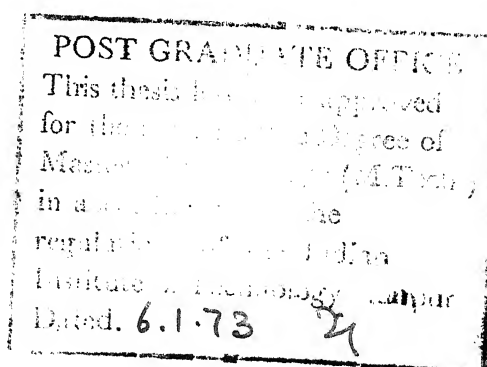


CERTIFICATE

This is to certify that the present work, "Studies in Viscosity of Rapidly Settling Suspension" has been carried out under my supervision and that this has not been submitted elsewhere for degree.

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Author

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ABSTRACT

The relevance of viscosity of rapidly settling suspensions to many industrial processes has been pointed out.

In the present study the effect of altering the surface characteristics by coating the particle surface, size-distribution, density and concentration of particles on apparent viscosity of their suspensions was investigated.

To undertake this study a sample container assembly was developed for viscosity measurement of rapidly settling suspensions. Viscosity measurements of suspensions upto a maximum concentration of 47% by volume of glass, alumina and quartz particles in various size-ranges in distilled were made in a Brookfield viscometer. All the measurements were taken at  $25 \pm 0.1^\circ\text{C}$ .

Particles were rendered non-wet by coating them with silicone oil. Reduction in viscosity upto a maximum of 20% was observed due to coating of glass at 20% volume concentration. A possible explanation has been given in terms of immobile liquid layer, electroviscous effects and interparticles friction.

The size-distribution of particles in a suspension was found to be significant in that when large proportions of fines were present, the viscosity appeared to be increasing with decreasing average particle size. But suspension consisting of mainly coarser particles resulted in increase of viscosity with increase in particle size.

The density of particles was also found to influence the viscosity. Heavier particles resulted in higher viscosities.

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## CHAPTER 1

### INTRODUCTION

The knowledge of rheological properties of rapidly settling suspensions is of great importance to material handling in chemical processes, transportation of solids as suspensions in pipe lines and in prediction of power requirements in agitation.

If the viscosity of suspensions is some-how reduced keeping a constant concentration, the power required for transportation of slurries through pipes will be correspondingly reduced rendering hydraulic transportation economical.

Generally, suspensions exhibit non-Newtonian behaviour as compared to Newtonian behaviour of the suspending media. Their behaviour is influenced by several factors which include size, shape, surface characteristics, size-distribution, concentration and density of the solids and viscosity of the suspending medium.

Although a lot has been reported about the influence of variables, like particle concentration, size, shape, size-distribution and shear rate on the viscosity of suspensions, various kinds of surface effects e.g. solid-liquid interactions have not been taken into account. Furthermore results from different sources are not consistent with theory and with each other.

### Literature Review:

The first attempt to treat mathematically the viscosity of a dispersed system is due to Einstein<sup>(1)</sup> who considered a suspension of rigid spheres in a viscous liquid. On the assumption that the aggregate volume of spheres was small as compared with that of liquid and that the spheres were sufficiently far apart not to influence one another, he arrived at the following equation:

$$\eta/\eta_0 = \eta_r = 1 + KC \quad (C \rightarrow 0)$$

where

$\eta_r$  = Relative viscosity of suspension

$\eta$  = Viscosity of suspension

$\eta_0$  = Viscosity of suspending medium

$C$  = Volume of solids per unit volume of suspension

$K = 2.5$ , (a constant)

It is of interest to note that the particle size, particle size distribution and particle number density do not appear in this equation. It is the total volume of solids that appears in the equation and affects the viscosity increase.

Vand<sup>(2)</sup> studied the influence of walls and interaction of particles for rigid, non-solvated system and derived the following correlation in the form of a power series:

$$\eta_r = 1 + 2.5 C + 7.17 C^2 + 16.2 C^3 + \dots$$

Results of viscometric experiments were compared with above equation and satisfactory agreement was found upto a concentration of 25% of particles by volume.

Robinson<sup>(3)</sup> attempted to extend the Einstein's equation introducing concept of free liquid. He deduced the following correlation:

$$\eta_{sp} = \frac{\eta - \eta_0}{\eta_0} = \frac{KC}{1-S'C}$$

where

$S'$  = Volume of sediment when particles themselves occupy unit volume (specific volume of sediment)

$C$  = Volume fraction of solids

$(1-S'C)$  = Volume of free liquid

= Total volume - affective volume of solids.

$K$  = A constant

He made the assumption of "packed sediment" made at high concentration when viscosity becomes infinite and there is not sufficient liquid to fill the voids between the packed particles. Specific volume of packed sediment depends upon the particle-size distribution and the degree of aggregation. Values of  $S'$  and  $K$  can be obtained by actually plotting  $C/\eta_{sp}$  versus  $C$ .

Mooney<sup>(4)</sup> extended the work of Einstein upto finite concentration for mono and polydispersed suspensions of smooth spheres. Mono disperse suspension is one where particles of uniform size are used. In poly disperse case size distribution

is present. He based his theory on the 'Geometric Crowding Action' between spheres and derived following equations:

$$\eta_r = \exp \left( \frac{2.5C}{1-KC} \right) \quad \text{for monodisperse}$$

$$\eta_r = \exp \left( 2.5 \sum_{i=1}^n \frac{C_i}{1 - \sum_{j=1}^n \lambda_{ji} C_j} \right) \quad \text{Poly disperse}$$

where

$K$  = self crowding factor;  $1.35 < K < 1.9$

$\lambda_{ji}$  = crowding factor of  $j$ th particle with  $i$ th particle.

He showed that the theory agreed well with best published data over the whole range of concentration between 0-50%.

Ting and Luebbbers<sup>(5)</sup> proposed the following model for free flowing spherical particles true for any concentration.

$$\frac{C_v}{\eta_{sp}} = C_{v\infty} - C_v$$

where  $C_{v\infty}$  is the volume fraction of solids when all the liquid is occluded in particle packing and is no longer free. Values of  $C_{v\infty}$  can be obtained by knowing the packing structure.

For poly disperse systems he modified above equation on the assumption that in this case smaller particle can fill the voids thereby changing the type of primary packing.

$$\frac{C_v}{\eta_{sp}} = (C_{v,\infty})_m - 0.79 C_v$$

where  $(C_{v,\infty})_m$  is the  $(C_{v,\infty})$  for the corresponding monodisperse system.

Moreland<sup>(6)</sup> undertook his study in connection with coal-transportation and studied the effect of particle size and size-distribution on viscosity of suspensions in a white mineral oil and concluded the following:

1. Slurries of smaller particles have higher viscosities than those of larger particles.

2. Slurries of irregular particles have higher viscosities than those of spherical particles.

3. A great decrease in viscosity is obtained by excluding all particles in middle size-range and using slurry consisting of 60-70% large particles together with 30-40% particles smaller than 1/10th the size of the larger particle.

Ward and Whitmore<sup>(7)</sup> investigated the effect of size distribution for spherical particles. They made the following conclusions:

1. Relative viscosity of suspensions is independent of:

- (a) Rate of shear upto 30% particle concentration

- (b) Absolute sizes of the spheres

- (c) Viscosity of suspending liquid.

2. Relative viscosity goes on decreasing with increasing size upto a constant value.

3. The proportionality constant between specific viscosity and volume concentration at infinite dilution were shown to be varying from about 4.0 for size ratio 1:1 to 1.9 for ratio exceeding 3:1.

4. Viscosity of irregular particles is higher than that for spherical particles.

Clarke<sup>(8)</sup> disagreed with the older work of Moreland and found that viscosity increased with increase in particle size for irregular particle. More the shape deviated from spherical higher viscosities were observed. He also observed reduction in viscosity of coarse settling suspensions by addition of 'Stereogene', a detergent, on a moderate concentration ( 0.1%) in water such that the addition does not change the viscosity or density of water significantly. In case of glass spheres addition of 0.6% by volume of 'Stereogene' gave rise to 10% reduction in viscosity at 40% volume concentration. He explained this decrease in terms of inter-particle friction which was claimed to be reduced by the addition of detergent in the suspension.

Purohit and Roy<sup>(9)</sup> showed that irregularities of particle shape increases the viscosity but this increase can be counter-acted by the factors like size-distribution and surface characteristics etc.

Broughton and Windelebank<sup>(10)</sup> investigated the effect of agglomeration on viscosity of suspension and found it to be higher in case of finer particles due to increase in attractive forces.

Many workers<sup>(11,12,13,14)</sup> have taken into account the effect of zeta potential on viscosity of suspensions. Viscosity

is greatly affected by addition of electrolytes thus altering the attractive and repulsive forces between particles and the liquid.

The prime objective of the present work was to investigate the effect of solid-liquid interaction, size-distribution, density and concentration of solid particles of glass, quartz and alumina on viscosity of suspensions in water. For this following type of work was undertaken.

1. Development of a Container Assembly to study the rheological properties of suspensions with fast settling solids.

2. Measurement of apparent viscosity for different concentration of glass, alumina and quartz particles of various size and size-distribution. The surface of glass and quartz particles was altered by coating them with silicone oil. The coated particles were found to be water repellent as confirmed by their contact angle of more than  $90^\circ$ . 'Capillary Rise Technique' was employed for measurement of contact angle (Appendix B). Viscometric data were taken for coated and uncoated particles. Further a mixture of 50% coated plus 50% uncoated quartz particles was used for viscometric measurements to observe the effect of partial wetting of particle on viscosity of suspensions.

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## CHAPTER 2

### EXPERIMENTAL SET-UP

Experimental set-up consisted of Measurements of

1. Viscosity of suspensions
2. Size-distribution of particles
3. Contact angles of glass and quartz with water.

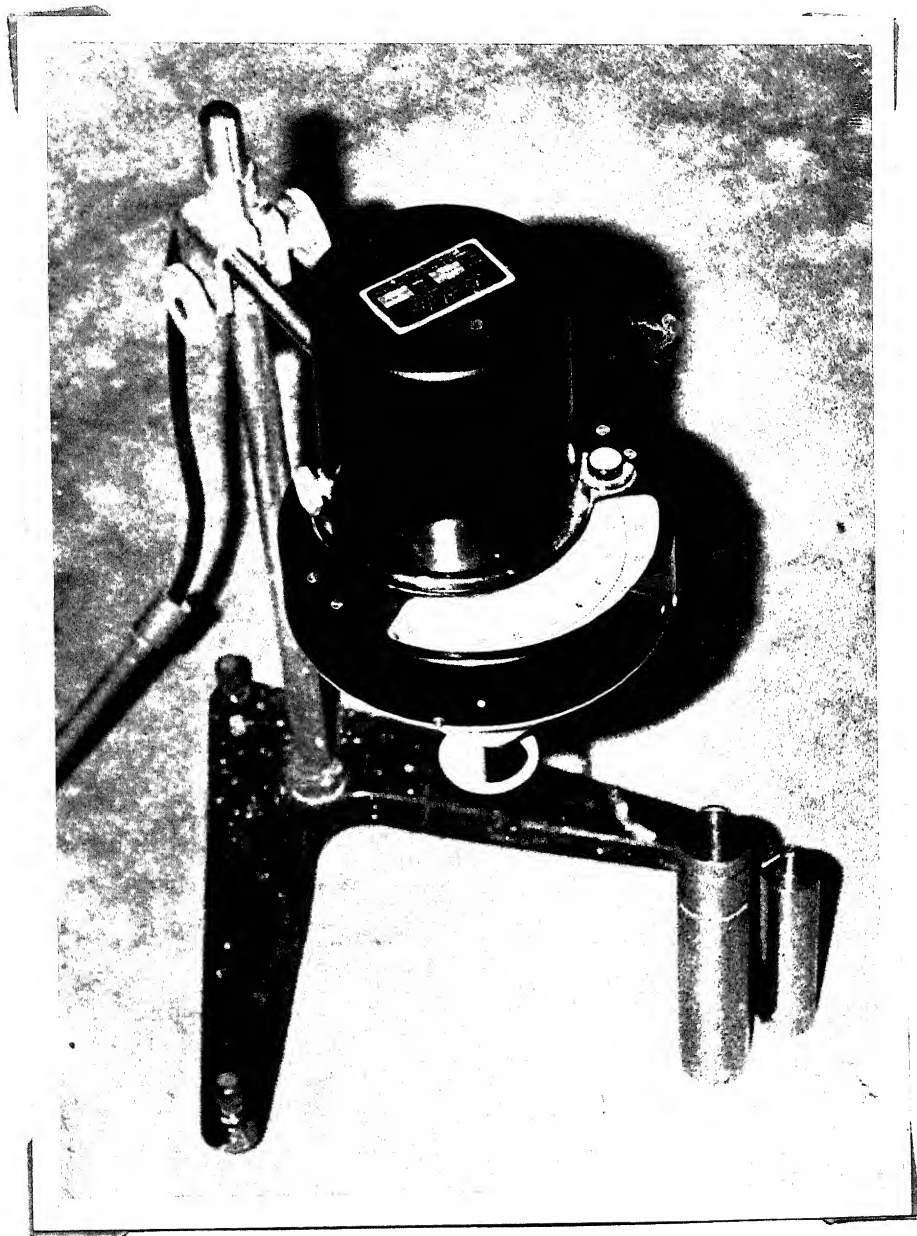
The descriptions of size-distribution and contact angle measurements have been discussed in detail in Appendices, whereas the set-up used for viscosity measurement has been described below:

#### Brookfield Model Synchro-Electric Viscometer<sup>(15)</sup>:

This viscometer (Figure 1) rotates a "spindle" in the fluid and measures the torque to overcome the viscous resistance to the induced movement. The spindle is driven through a beryllium copper spring - the degree to which the spring is wound, indicated by the position of the red pointer on the viscometer dial, is proportional to the viscosity of the fluid for any given speed and spindle. The accuracy of  $\pm 1\%$  is guaranteed by the manufacturer.

#### Brookfield U.L. Adapter:

As the desired viscosity was within 1-10 cp range, the Brookfield L.V.F. viscometer was employed in conjunction with U.L. adapter. This arrangement allows the viscosity measurement over a range of 0-10 cp with extreme precision and accuracy. The reproducibility of  $\pm 2\%$  has been reported by the manufacturer.



*Fig. 1 - Brookfield viscometer with tube and spindle.*

The U.L. adapter arrangement also eliminates turbulent flow in the low viscosity range as well as ensures a greater sensitivity. The following procedure is necessary for installing the U.L. adapter.

The spindle coupling nut is screwed onto the mating viscometer coupling screw. The main adapter body (B) is then inserted over the pivot housing (A) and turned until it snaps flush against the bottom of housing. The clean spindle (C) is then attached by the hooking arrangement as shown in Figure 2. The clean tube (D) is placed over the spindle and attached in the adapter by pushing it up until horizontal groove in the tube coincides with the screw. Slight rotation to left or right then fixes the tube in its position. The viscometer is then operated in its normal fashion.

Brookfield Laboratory Stand Model A:

It consists of a special clamp which will securely hold the viscometer. The clamp is the part of a rack and pinion system which enables the viscometer to be quickly raised or lowered to a precise point. Thus, the viscometer spindle can be immersed to the correct depth with minimum of trouble.

The heavy cast iron base of stand is provided with three screws for easy setting of the viscometer level.

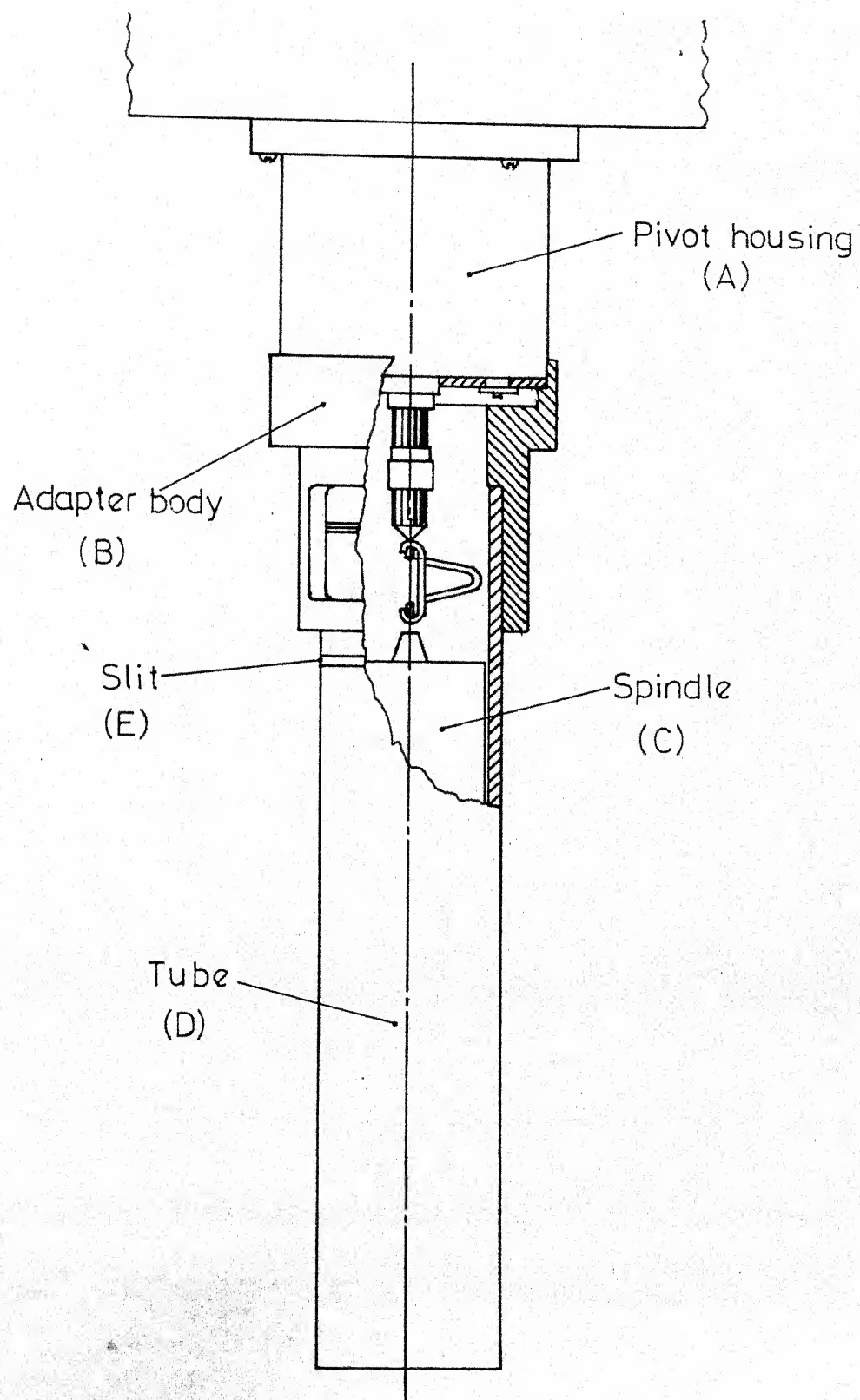


Fig.2

Sample Container Assembly:

To measure viscosity of rapidly settling suspensions, a container assembly was constructed, similar to the one as suggested by Clarke<sup>(8)</sup>. It consists of a stirrer rotated through a shaft by a motor and a container with baffles, (Figure 3). The main body of the container was made up of perspex so that the suspensions would be visible. Diameter and depth of the container was decided so as to accommodate the U.L. adapter as follows:

Depth : 17 cm.  
Inner diameter: 5.5 cm.  
Clear dia : 3.5 cm.  
(after baffles)  
U.L. adapter tube  
outer diameter : 3.0 cm.

To avoid turbulence, six baffles were provided along the wall of the container. The angle of the stirrer blade was kept such that the suspensions rose along the baffles and part of it entered in the U.L. adapter tube, specially made for this purpose with three slits of about 2 mm width, covering nearly 90% of the periphery of the wall. The tube was open at the bottom so that suspension could flow down the tube and then along the sides of the baffles, thus maintaining a steady circulation. But this gave rise to undesirable vortex formation on the bottom end of the spindle which was quite near to the stirrer. This difficulty was overcome by fixing a disc on the baffles as shown in

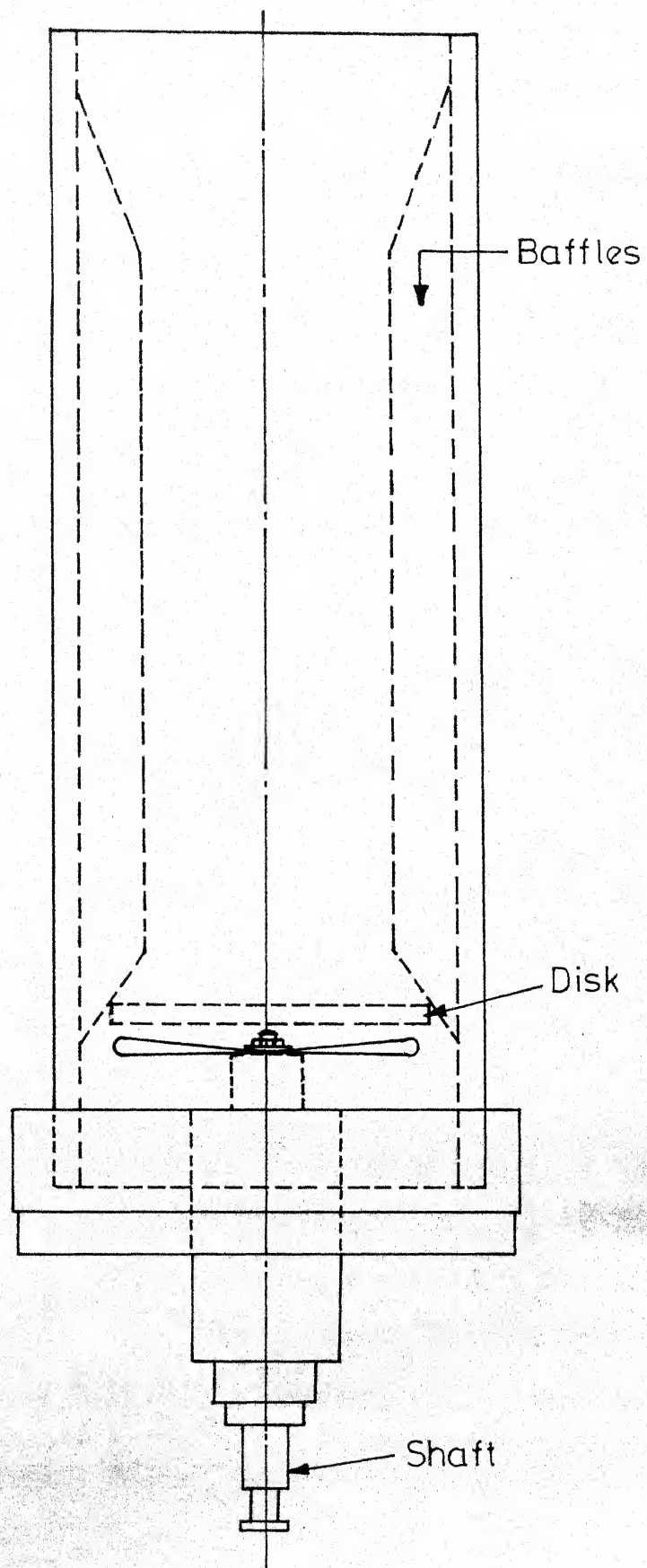


Fig. 3

figure. Such an arrangement avoided the turbulence as well as vortex formation in the tube. To ensure reproducibility of data, the speed of the stirrer was kept constant at 600 R.P.M. for all measurements. This was done by varying the voltage to the motor of the stirrer by means of a variac and checking the speed with stroboscope.

The uniformity of suspension was checked by taking out the samples at different depths and different points along the diameter with the help of a pipette. The variation were within  $\pm 5\%$  and in the zone of interest it was even less. For stirrer speed of less than 600 R.P.M. this uniformity could not be obtained.

The viscometer with this new assembly was calibrated with distilled water and with standard viscometric liquids.

#### Water Bath:

All measurements were carried out at  $25^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ . by using a constant temperature water bath, model - ultra-thermostat type NBE. This consists of a heating device, built in stirrer, pump and a thermostat, controlling temperature between  $\pm 0.02^{\circ}\text{C}$ . Constant temperature water from this bath ~~was~~ circulated in the auxiliary water bath in which the sample container assembly was fitted.

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## CHAPTER 3

### EXPERIMENTAL PROCEDURE

#### Material Used:

Table I

Sl. No.	Solids	Mesh Size	D <sub>Average</sub> (Microns)	Method of Size Measurement
1.	Glass Sphere	-170 + 230	74	Sieving through ASTM sieves
2.	Glass Grounded	-230	37	Sedimentation method
3.	Quartz "	-200 + 270	63	Sieving through ASTM sieves
4.	Quartz "	-270	25	Sedimentation Method
5.	Alumina Grounded	-200 + 270	63	Sieving through ASTM sieves
6.	Alumina Grounded	-400	14	Sedimentation Method

The grain size analysis of serial no.2,4 and 6 has been tabulated in Table II A through IIC.

#### Sample Preparation:

The quartz and alumina particles (-200 + 270) were prepared by grinding coarser particles in a ball mill and sieving them into desired size range (ASTM sizes). Alumina (-400 mesh) and glass spherical particles (-170 + 230) were used as such from the suppliers\* after ascertaining their size and size-distribution.

\*Source of supply: Alumina Ceramic Lab. IIT-Kanpur .  
Glass Epine Scientific Co., U.S.A.



TABLE II AGRAIN SIZE ANALYSISGlass D<sub>avg</sub> : 37 Microns

Weight Percentage	Finer than, microns
95	65
75	60
50	33
25	16
10	8
5	5

TABLE II BGRAIN SIZE ANALYSISAlumina D<sub>avg</sub>: 14 microns

Weight Percentage	Finer than microns
99	40
75	26
50	7
25	1.5
5	1

TABLE II CGRAIN SIZE ANALYSISQuartz D<sub>avg</sub> : 25 microns

Weight Percentage	Finer than Microns
95	55
75	37
50	25
30	10
5	6

### Cleaning Surface of Particles:

The particles were first washed thoroughly with Acetone and then immersed in hot chromic acid for 18 hours. The acid traces were removed by rinsing the particles with distilled water for a number of times.

### Coating the Particles Surface:

To study the effect of wetting characteristics of particle on viscosity of their suspension, the particle surface was coated with silicone oil\*.

Appropriate amount of silicone oil<sup>†</sup> was dissolved in Acetone (AR grade). Glass particles (-170 + 230) were poured into this solution allowing them to be completely immersed. The slurry was thoroughly stirred with glass rod to ensure uniform coating on all the particles. The beads were left immersed over a sufficient time for adsorption of silicone oil on glass surface. The supernatant liquid was drained and Acetone was evaporated at room temperature. Dried particles were found to be completely non-wetting with water.

Same procedure was adopted for coating quartz powder with silicone oil.

When such coated particles were added to distilled water, they formed clusters together and did not settle down. This was

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\*L-45 Silicone; viscosity 350 Cstks; supplied by Union Carbide Corporation, Chemical & Plastics, New York, 10017, U.S.A.

<sup>†</sup> 0.5 percentage by weight of solids

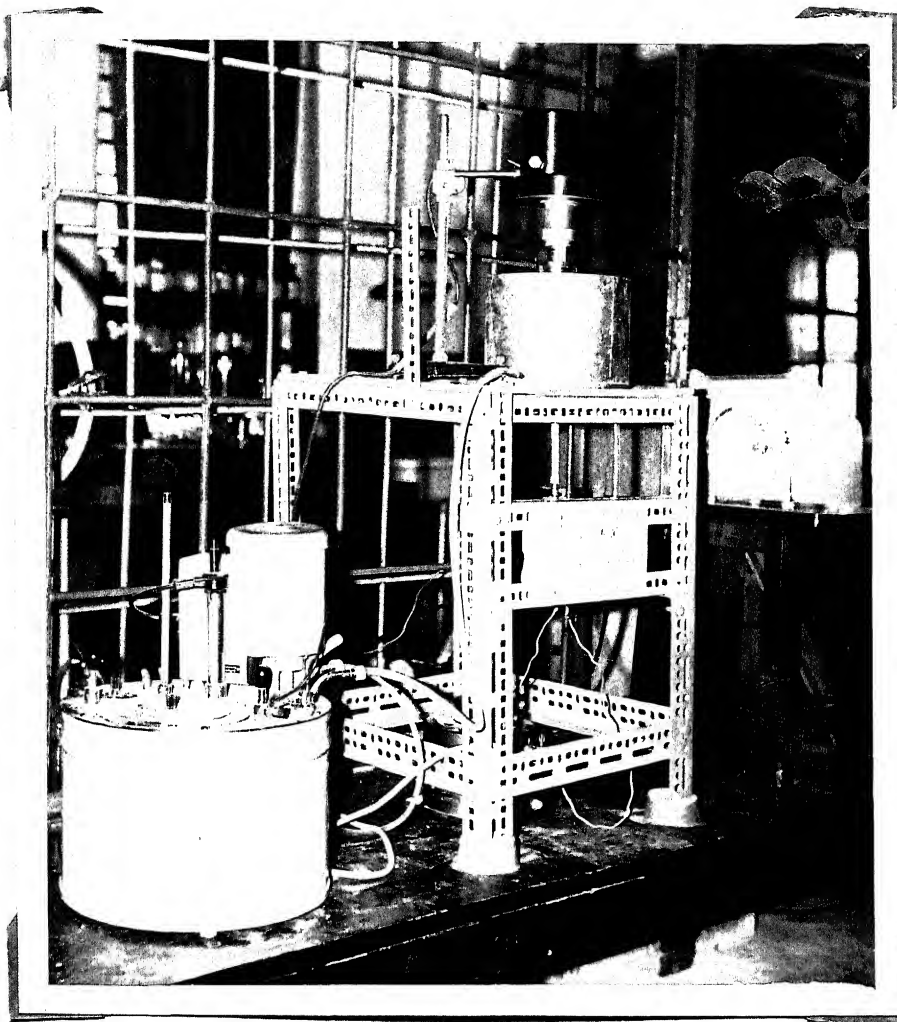
due to the air sticking around the particles. Vigorous shaking allowed them to settle down but still they were not completely dispersed. Hence, the system was deaerated by heating and by centrifuging till the air responsible for cluster formation was completely released, and particles settled down like uncoated particles. A portion of supernatant liquid was tested for viscosity which matched with the viscosity of distilled water indicating that supernatant liquid did not carry any dispersed silicone oil.

#### Viscosity Measurement (15):

For viscosity measurements the suspensions of desired concentration were prepared in 300 ml of water. Water circulation in auxiliary bath was started from the main constant temperature bath, which was maintained at  $25 \pm 0.02^\circ\text{C}$ , Figure 4. The motor of the stirrer was turned on and suspension was added to the container. Viscometer was levelled on the platform while its spindle and tube were immersed in the suspension upto a level such that slits on the tube were just immersed within suspension.

After a steady-state temperature was obtained, viscosity was measured at spindle speed of 60 R.P.M. Three to four repetitions of measurements were made and average of these was recorded as the viscosity of suspension at that setting.

Viscosity at different shear rates was not measured due to experimental limitations, so it was not possible to obtain



*Fig. 4 - Experimental Set-up.*

behaviour of suspensions with different shear rates. All the measurements were made at a constant spindle speed of 60 R.P.M. and the reported viscosity was indeed apparent viscosity at that shear rate.

A sample data collection has been inserted in the Appendix C. Fig. 5 is the calibration curve for present set-up which shows the observed value of viscosities of standard liquids against their standard viscosities. All the plotted viscosity data in the various figures are the corrected value of viscosities taken from this calibration curve.

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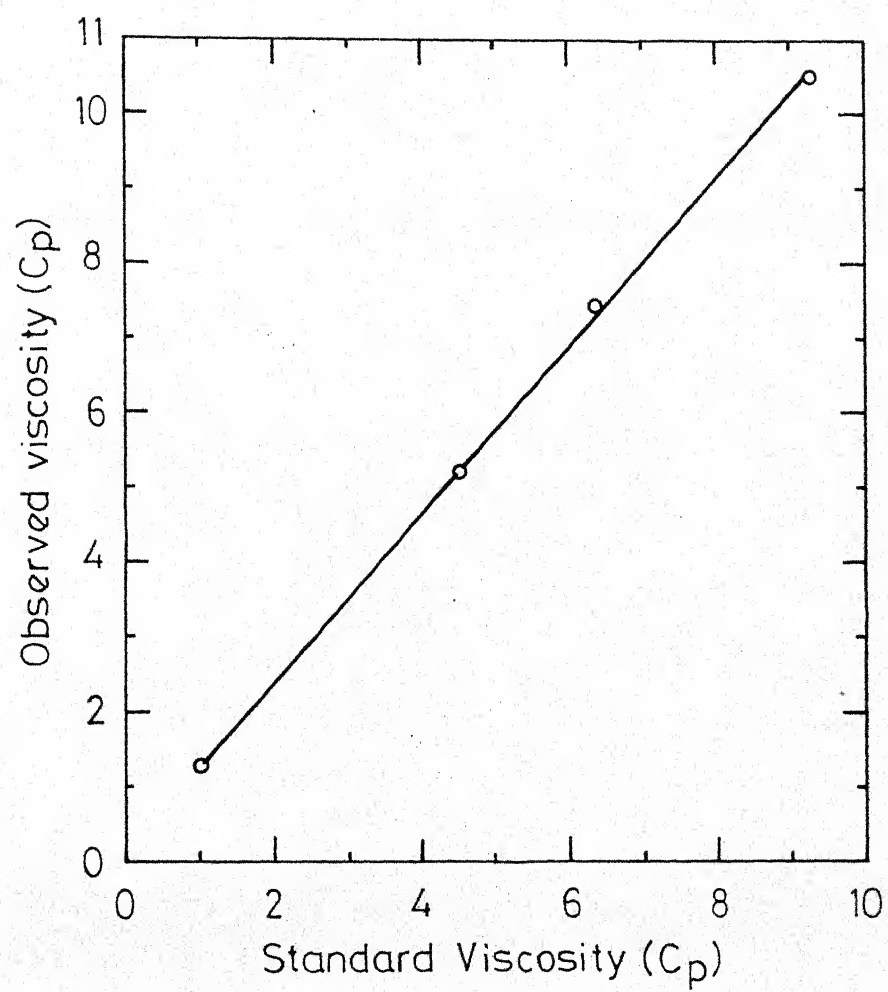


Fig. 5 - Calibration Curve.



## CHAPTER 4

### RESULTS AND DISCUSSION

The viscosity of suspensions as function of concentration of solid particles have been plotted in Figures 6A to 8. Interpretation of the results and discussion on the effect of various variables studied, is presented below:

#### (a) Effect of Particle Concentration:

The increase in relative viscosity of suspensions with increase in concentration is evident from all the viscosity - concentration plots. However, this increase with solid concentration has been found to be dependent upon various parameters including particle density, size, size-distribution and solid liquid interactions (Figures 6A to 8).

Normally, viscosity concentration relationship has been found to be linear in the entire range of concentration studied when the particles were of uniform size. However, in case of alumina and quartz higher increase in viscosity has been observed beyond a certain transition concentration more particularly in presence of size distribution. This transition point has been found to vary according to the type of solid used and extent of size distribution. Below this point the viscosity increases slowly as a function of flow around discrete particles. Above this point the solids have no room to flow freely and undergo a series of impacts giving rise to a much higher rate of increase in viscosity with concentration.

(b) Effect of Particle Coating by Silicone Oil:

As can be seen from the viscosity concentration relationship plots for glass and quartz (Figure 6A to 6C) the suspensions of coated particles in water have a lower viscosity than those of the uncoated particles. This reduction in viscosity for various cases has been tabulated below:

TABLE IIIREDUCTION IN VISCOSITY DUE TO COATING OF PARTICLES

Material	D <sub>avg</sub> Microns	Vol.%	Extent of coating*	% Reduction in Viscosity
GLASS	74	20	Complete	18%
GLASS	37	20	Complete	145%
QUARTZ	63	20	Complete	10
QUARTZ	63	40	Complete	14
QUARTZ	63	20	Partial	6
QUARTZ	63	40	Partial	9

\*Uncoated particles were water-wet and showed a contact angle of less than 5° whereas particles coated with silicone oil had the contact angle greater than 90°. A mixture of the uncoated and coated particles in equal proportions gave the partially coated samples. Measurement of contact angle is detailed in Appendix B.

The possible interpretations for this reduction are discussed below:

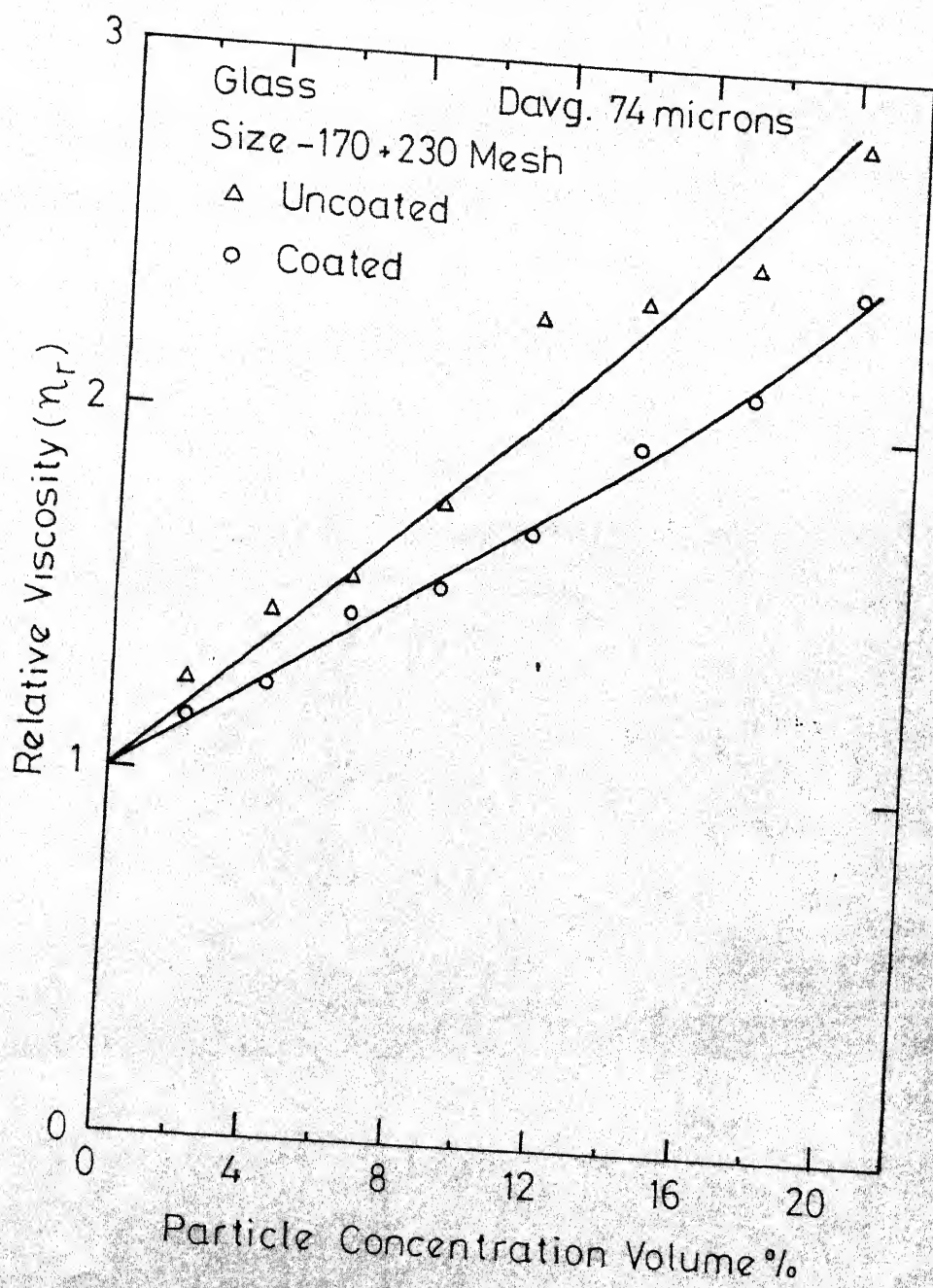


Fig.6A - Effect of Silicone Oil Coating of Particle on Viscosity of Suspensions.

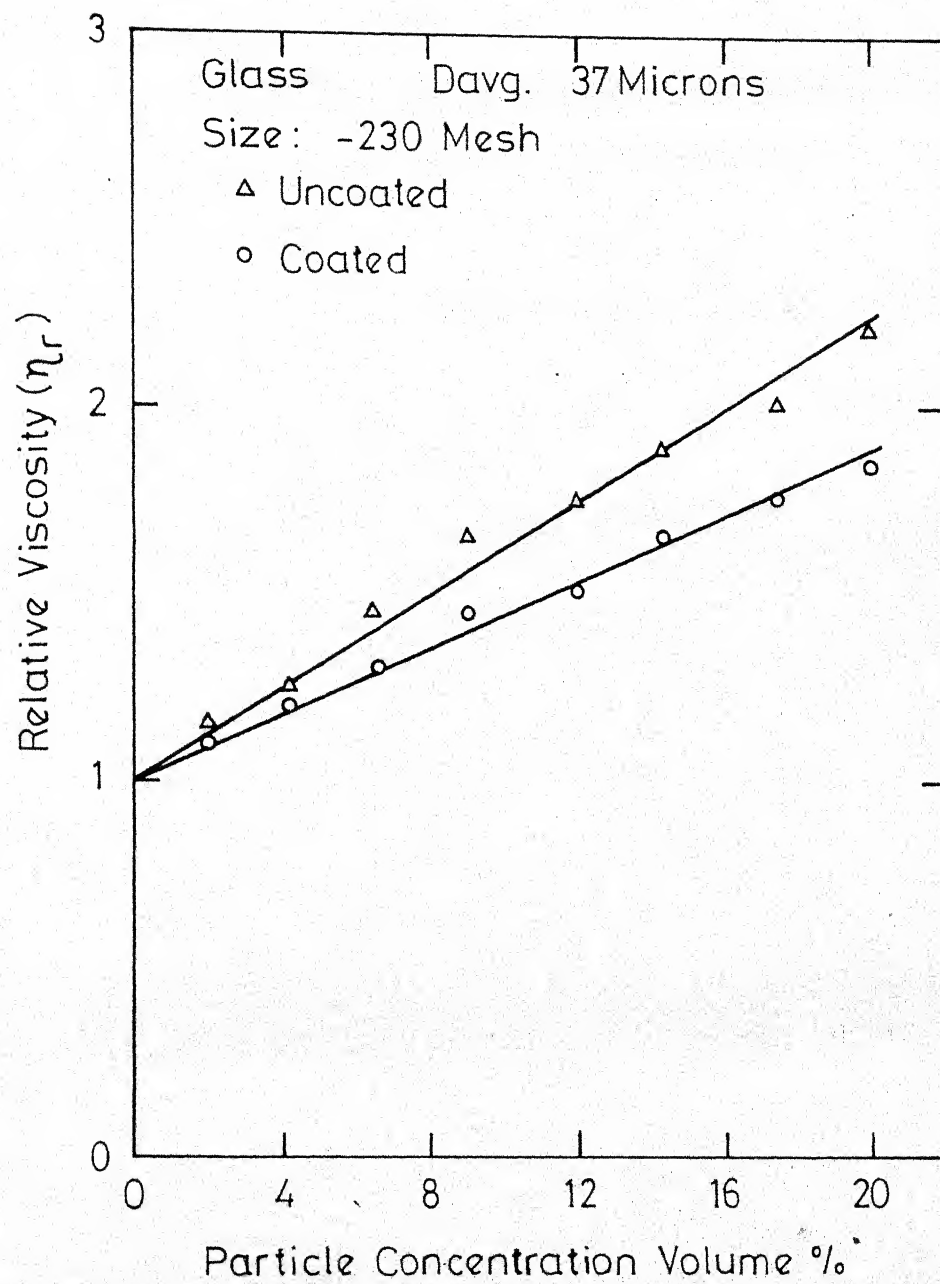


Fig.6B- Effect of Silicone Coating of Particles on Viscosity.

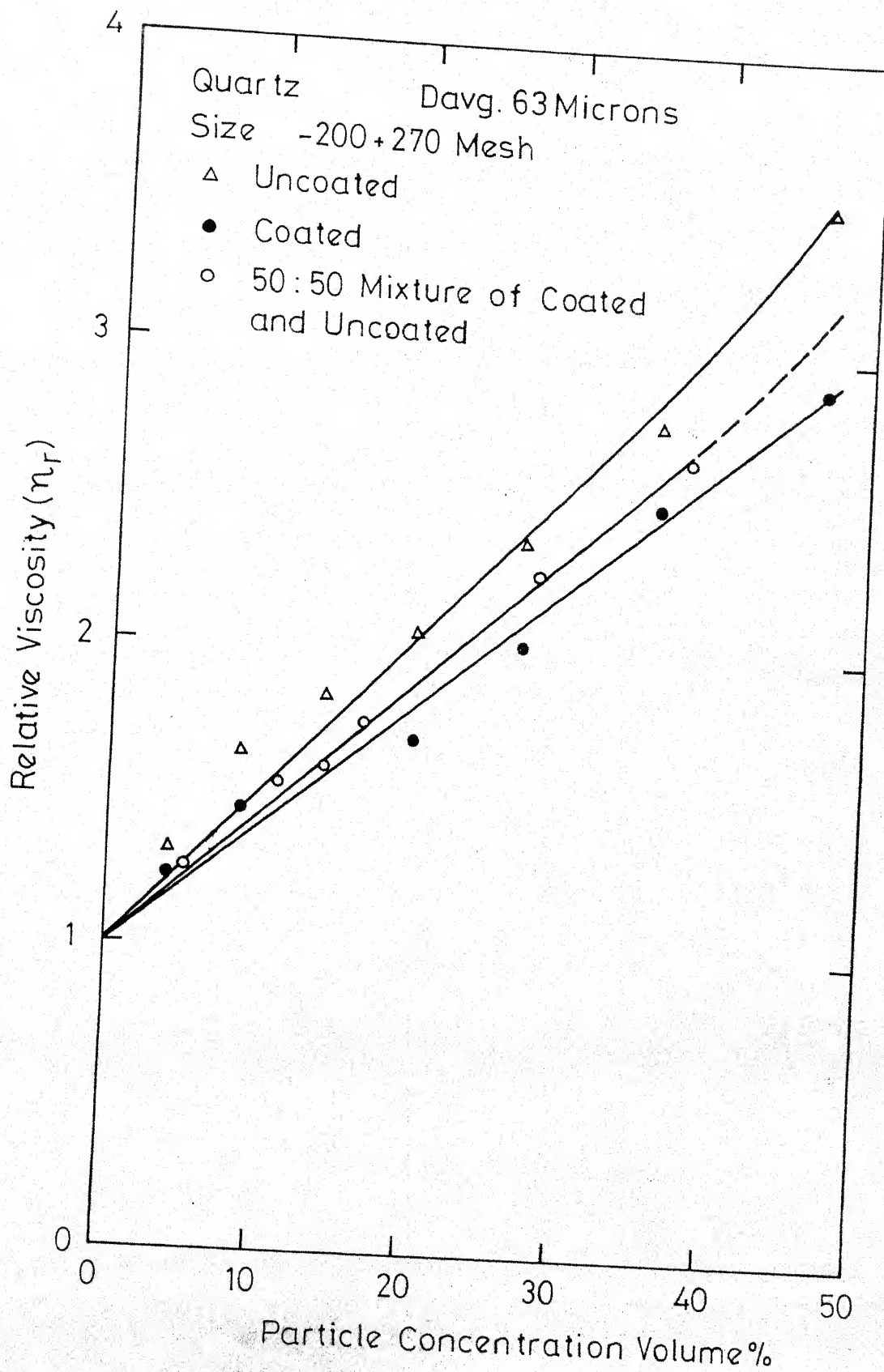


Fig.6C- Effect of Silicone Coating of Particle on Viscosity.

1) Immobile Layer Theory: It has been widely reported in literature that the presence of long range unbalanced forces on the solid surface causes liquid molecules near the surface to get adsorbed on it. This results in the formation of a thin immobile layer around the solid particles which is in equilibrium with bulk of the same liquid. Drost-Hansen<sup>(16)</sup> has strongly supported this concept. Dergaguin et.al.<sup>(17)</sup> have reported the thickness of adsorbed films for several liquids on glass surfaces. This thickness may vary from 66°A to 1000 °A for glass in water, depending on the nature of solid-liquid interface. A reasonable assumption for the thickness of thin films in equilibrium with bulk liquids will be atleast of the same order of magnitude.

Singhal, et.al<sup>(18)</sup> have supported the general conclusion that film thickness depends on nature of solid liquid interface. It is low for low energy surface like teflon as compared to that of high energy surfaces like glass and quartz.

When the particles are coated they become non-wet with respect to water and thus behave as low energy solids. Hence film thickness is low for the coated particles whereas uncoated particles due to their polarity are high energy-substances, hence are attached with immobile layer of significant thickness adjacent to the surface.

This immobile layer on the particle surfaces increases the effective volume of the solid particles in suspensions, increasing the apparent viscosity when these surfaces are water wet.

ii) Electro Viscous Effect: According to Conway and Du  
Duclaux<sup>(13)</sup> when charged particles are suspended in water, they  
give rise to the formation of an electrical double layer.  
Spherical symmetry of this double layer is distorted when the  
suspension is made to flow. This results in extra dissipation  
of energy because of the disturbances of electrical interaction  
between the ions in the double layer and charge on the surface  
of the particle. This extra dissipation of energy leads to an  
increase in viscosity of suspensions.

It has also been reported extensively in the literature  
that the electroviscous effects depend largely upon the thickness  
of double layers, the zeta potential being the measure of it.

It has been investigated by Young<sup>(14)</sup> that surfaces  
like graphite and other low energy substances exhibit practically  
no electrostatic field because of their hydrophobicity. Hence,  
electroviscous affects are absent in this case.

This view clearly suggests that the viscosity of the  
suspensions made up of charged particles will exhibit higher  
viscosity than those made up of coated particles which are low  
energy particles having no charges on them.

iii) Inter Particle Friction: The nature of the surface  
of solid plays an important role in viscosity of suspensions.  
Sharp-edged, granular solids give rise to higher viscosities as  
compared to rounded grains and spheres. This may be partly  
due to immobile layer of inert liquid which increases effective

volume concentration. But it is also reasonable to assume that particles with irregular surfaces will interact severely and inter particle friction will cause the viscosity to increase significantly. Coating may provide a kind of lubrication to solid surface rendering it smoother, compared to uncoated particles. Less interparticle friction can therefore be expected in case of coated particles. Smoother surfaces will allow greater slip resulting in the lowering of viscosity.

Referring back to immobile layer theory, if film thickness is taken to be of the order of  $1000^{\circ}\text{A}$ , then it will cause a volume increase of nearly 13% for particles size of 5 microns. For smaller particles, the effect will be even more. This volume change can account for the major part of reduction in viscosity due to coating. However, if the particle size is about 50 microns, the change in effective volume with  $1000^{\circ}\text{A}$  thick film is about 1% which is not significant. In case the thickness is assumed to be only  $100^{\circ}\text{A}$  then even for 5 microns particles the film contribution is negligible. This indicates that the reduction in viscosity on coating can be explained in terms of immobile liquid layer only in case of particles smaller than 5 microns. Due to lack of knowledge about exact thickness it is not possible to quantitatively interpret the present data in light of immobile layer theory.

Hence, reduction in viscosity in case of coated particles appears to be mainly due to reduction in interparticle friction



and electroviscous effects.

(c) Effect of Particle Size and Size-Distribution:

Glass Suspensions: From Figure 7A it is clear that in case of glass suspensions in the average size range of 37 to 74 microns, viscosity increased with increase in particle size. Table 2A gives the size distribution for the particles with average size of 37 microns the range being from 65 microns to 5.0 microns. The other lot with average size of 74 microns was a close cut of -170 + 230 mesh size. This increase may be attributed to the following factors:

1. Increase of particle size: increases inertia, so they exhibit greater amount of interparticle friction consequently increasing viscosity of the system.

2. Glass being amorphous, larger particles will be more irregular and sharp edged than the finer particles.

3. From Table 2A it is seen that particles of average size of 37 microns contain only 10% of the particles finer than 8 microns. These small amount of fines can easily circulate in the voids between larger particles resulting in a reduction in the number of interparticle impacts giving rise to reduction in viscosity.

Alumina Suspensions: Referring to Figure 7B, the results for grounded alumina suspensions seemed to exhibit opposite trend compared to those by glass particles i.e. viscosity increased with decreasing particle size. There is an increase in viscosity

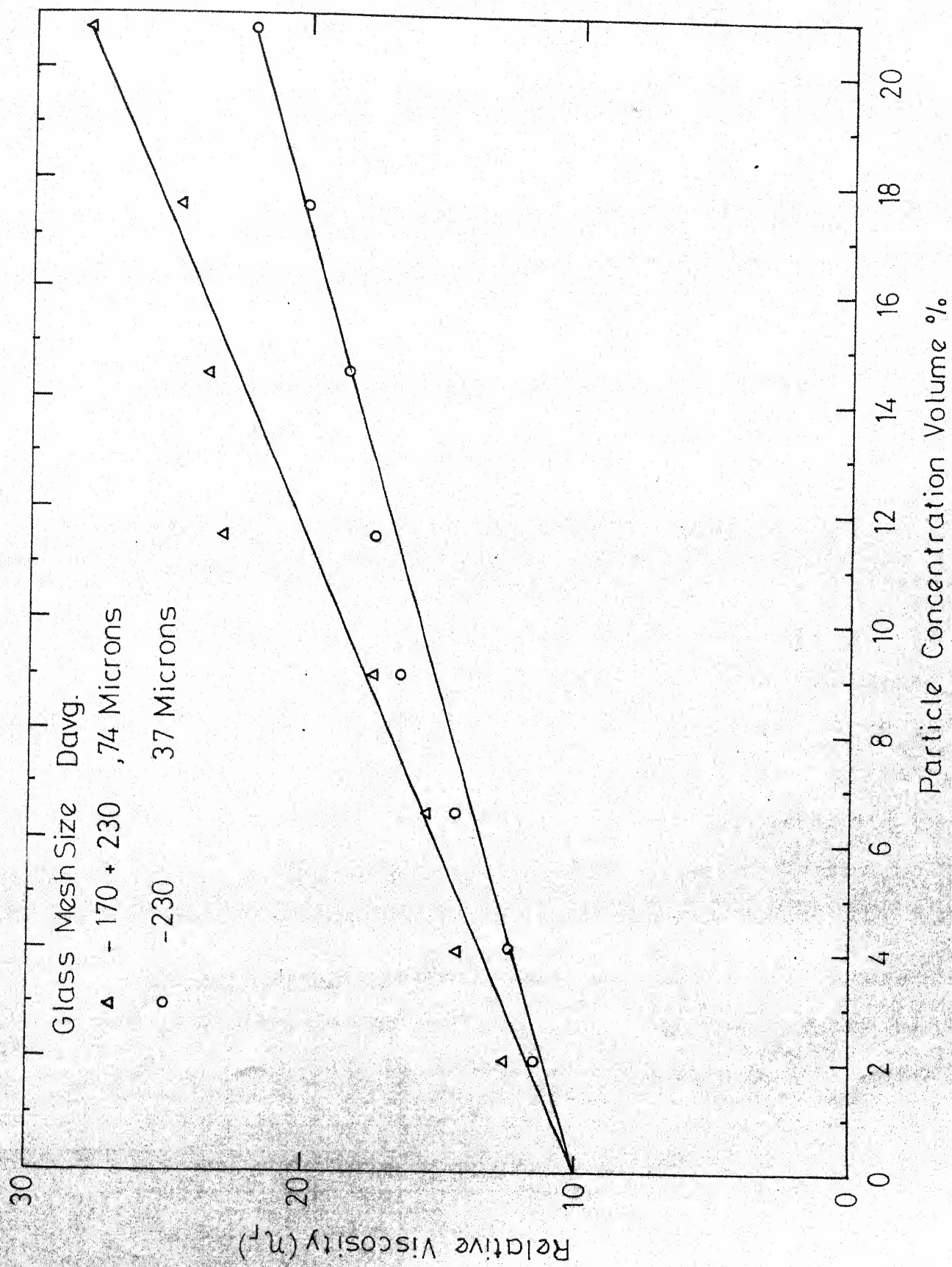


Fig.7A- Effect of Size - distribution on Viscosity.

of about 100%\* when the average size varied from 63 to 14 microns. Table II B gives the size distribution for the particles with average size of 63 microns in a close cut of -200 + 270 mesh size. The other lot with average size of 14 microns contained the particles in the range vary from ~~4.0~~ microns to 1.0 microns. This increase in viscosity can be attributed to large surface area of alumina particles. It may also be pointed out that beyond 12% volume concentration there is a steep rise in viscosity for the particles of average size of 14 microns. This may be due to the presence of as large as 50% of particles of size less than 7 microns. So, at higher concentration, the increase in volume due to immobile layer of water on the solid alumina surface will be very much significant as discussed earlier. Further, this large number of roughned fine particles at high concentration interlock themselves and scrap severely to yield higher dissipation of energy. Agglomeration effects should also be significant in case of fine particles.

Quartz Suspensions: For quartz suspensions the increase in viscosity with decreasing particle size has been found to be about 30%\* by varying the average particle size from 63 microns to 25 microns. This increase canbe explained in the same lines as has been done for alumina suspensions.

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\* At 20% volume concentration

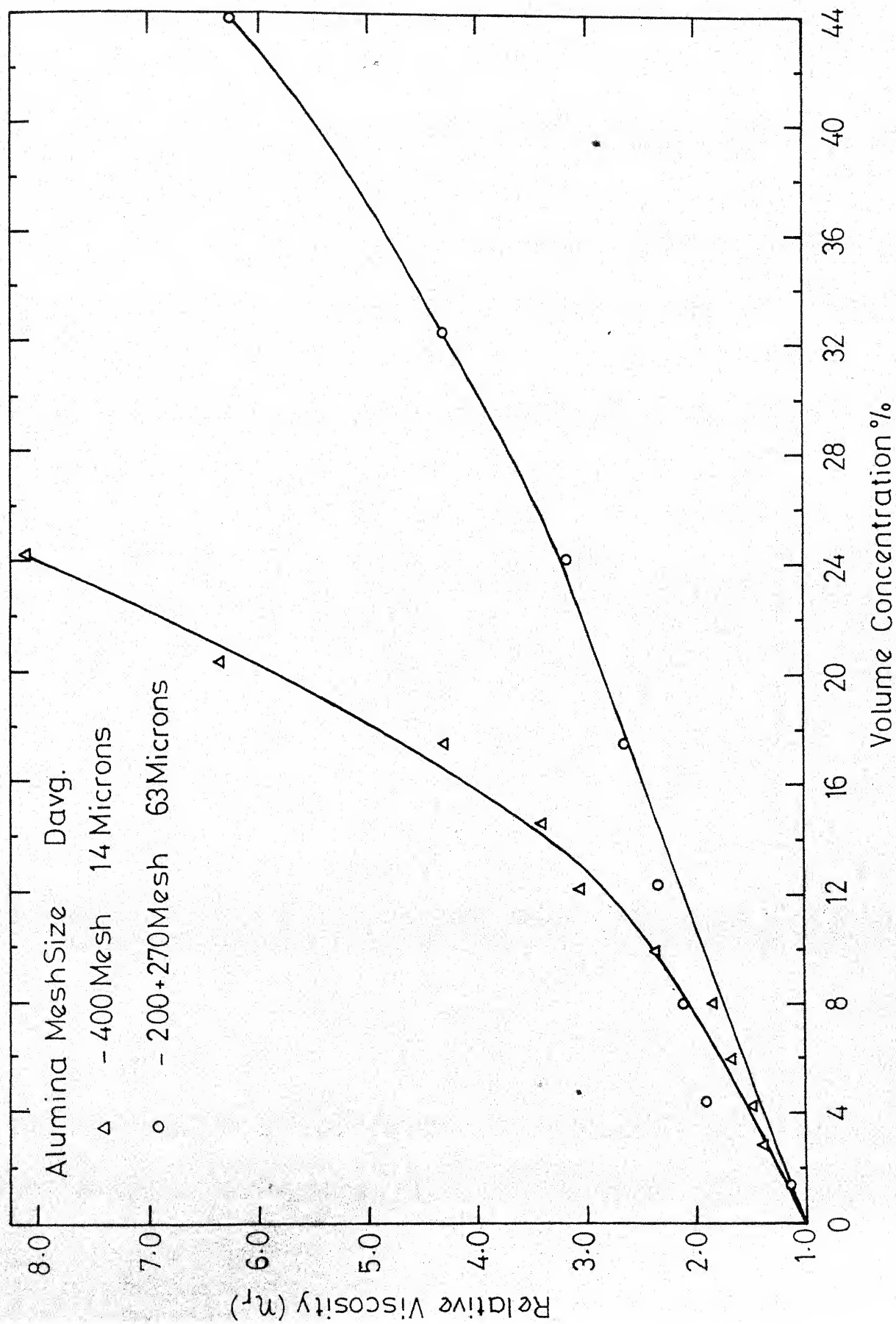


Fig.7B-Effect of Size -distribution on Viscosity.

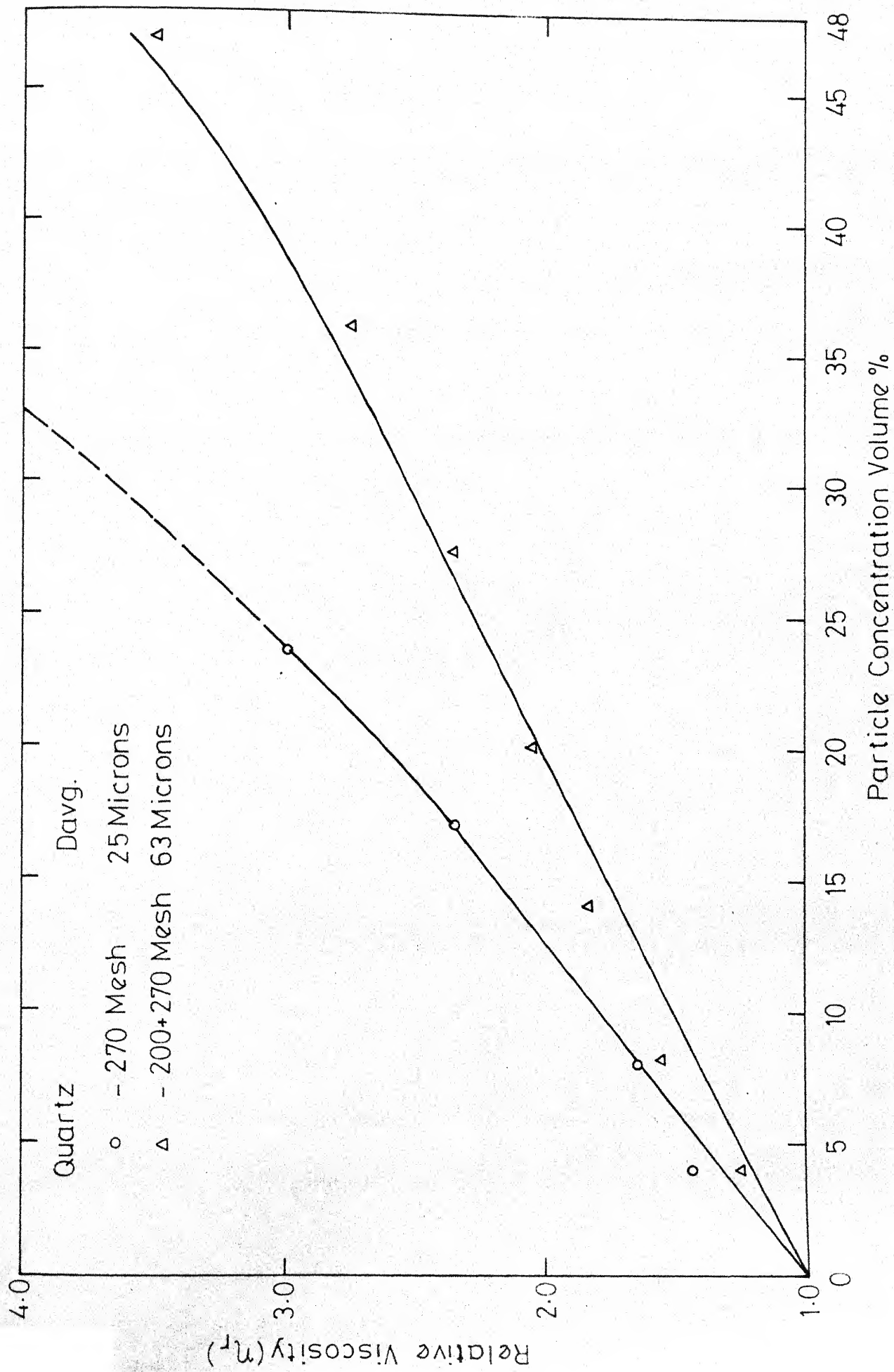


Fig.7C - Effect of Size - distribution on Viscosity.

(d) Effect of Particle Density:

Figure 8 shows that for a given volume concentration, alumina suspension has higher viscosity than quartz suspensions of same average particle size. Density of alumina is 3.9 whereas that of quartz is 2.65. This means heavier the particles, more is the viscosity of suspension. Hence, the influence of inertia of the particles on the viscosity is expected. Heavier particles can move with greater momentum and rebound from other particles. On interaction they are momentarily retarded and then accelerated; in both stages their inertia affects the amount of energy required. The dissipation of energy appears as extra viscosity.

However, as the alumina and quartz particles chosen for comparison of results are positively and negatively charged particles respectively, the difference in viscosity of their suspension can also be attributed to variation in their electroviscous effect. Nevertheless, inertia of the particles appears to be atleast partly responsible as is reported by Clarke<sup>(8)</sup> and Purohit, et.al.<sup>(9)</sup>.

Figure 8(A) shows the same effect of particle density on the viscosity of suspension as in Figure 8, except that concentration has been expressed in weight percent rather than volume percent. It is to be noted that in this figure the two curves for alumina and quartz are close to each other as compared to Figure 8.

This difference arises because in the case when volume is used for concentration the effect of density difference is clearly indicated. When weight is used for concentration density effect is eliminated and the remaining difference must be explained in terms of nature of surface charge and its density.



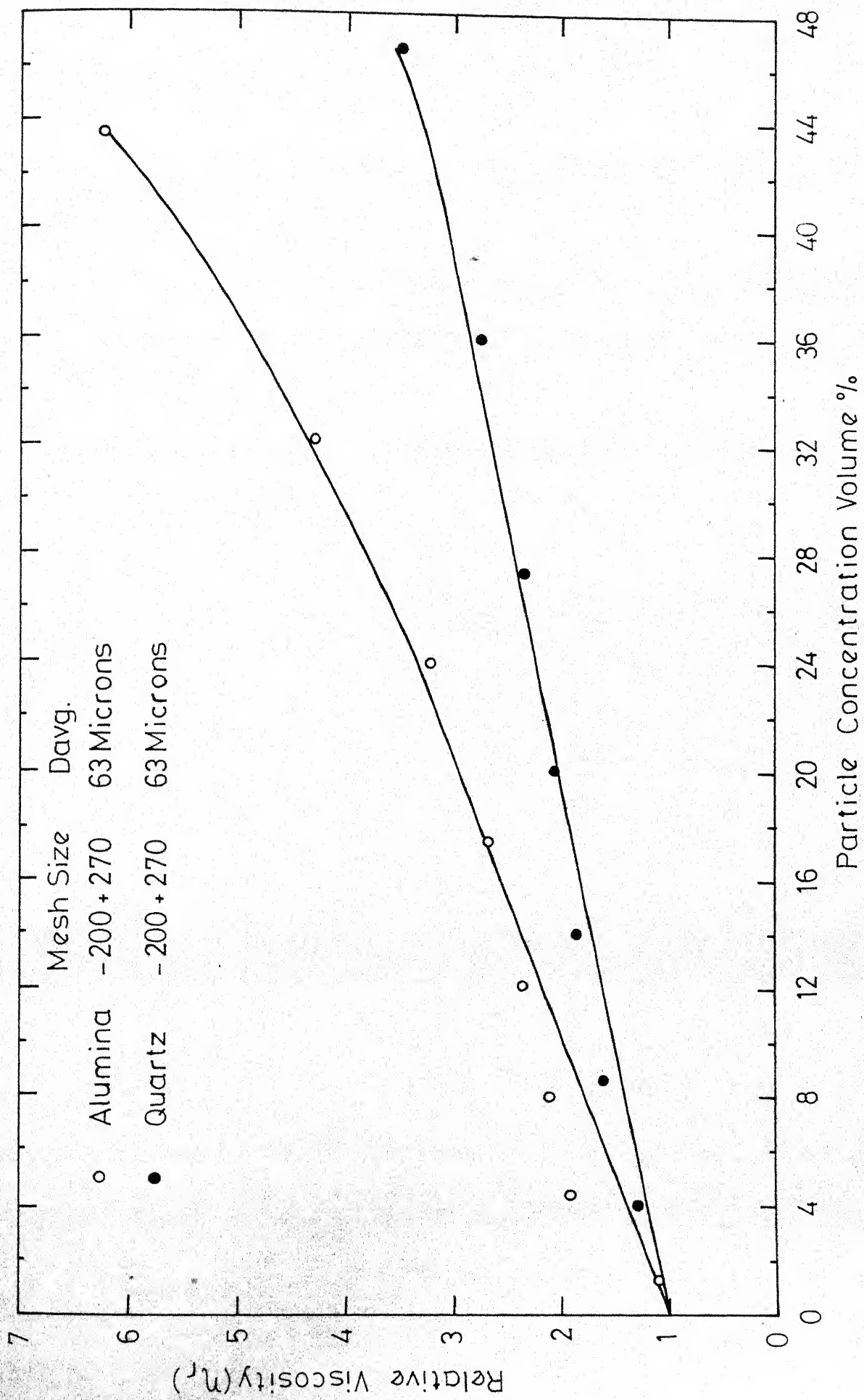


Fig. 8 - Effect of Particle Density on Viscosity.

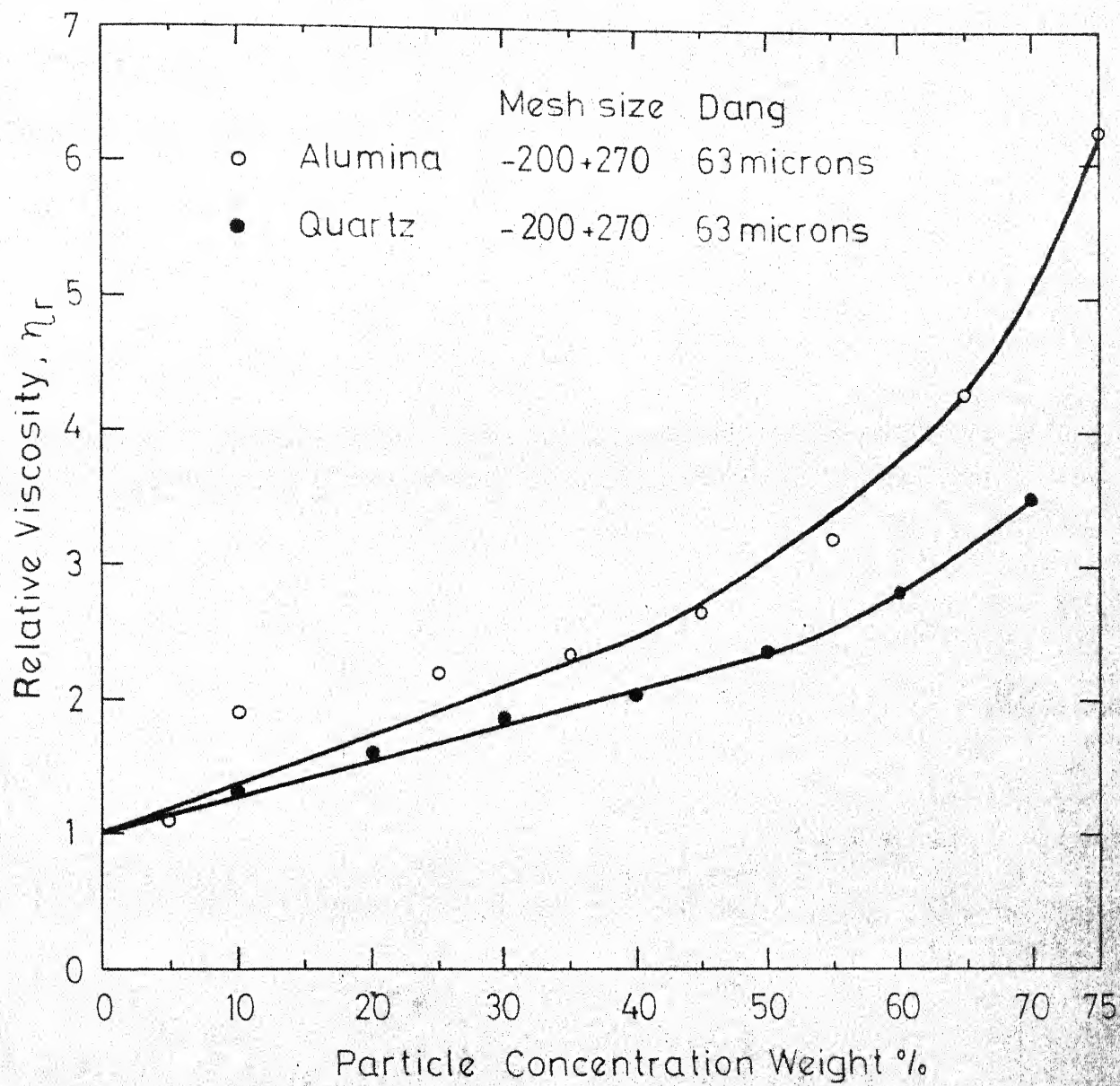


Fig.8A - Effect of particle density on viscosity.



## CHAPTER 5

### CONCLUSIONS

1. The sample container assembly developed for the study of viscosity of rapidly settling suspensions proved satisfactory upto volume concentration of 20 to 50 percent by volume of solids, depending on size of the particles.

2. Coating the particles surface with silicone oil resulted in reduction of apparent viscosity of suspensions significantly.

3. The apparent viscosity was found to vary almost linearly with concentration of particles in the entire range studied when uniform-size particles were used. However, size-distribution of solids affected the linearity. Higher viscosities with decrease in particle size were obtained when suspensions consisted of mainly fine particles. However, lower viscosity was yielded with decrease in particle size in case the suspensions were made of largely coarser particles and fewer fines.

4. Particles of higher density yielded higher viscosities, suggesting the possible effect of density of the particles on viscosity of their suspensions.

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## CHAPTER 6

### RECOMMENDATIONS

1. In the present study all measurements of viscosity were made at one shear rate only (60 R.P.M.) because of equipment limitations. Hence, study on shear rate dependency of viscosity of rapidly settling suspensions should be carried out to get a complete picture of their non-Newtonian characteristics.

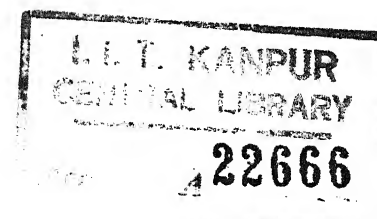
2. It was not possible here to quantitatively ascertain the effect of immobile layer on the particles in suspension because of lack of knowledge about its thickness. Hence measurement of immobile layer thickness should be taken up to ascertain its contribution on viscosity of suspensions.

3. Finally, more extensive studies are needed for investigating the effects of particle density and particle-size-distribution on viscosity in order to establish useful relationships.

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APPENDIX ASIZE-DISTRIBUTION ANALYSIS: HYDROMETER METHOD

The method describes a procedure for the quantitative determination of the distribution of sub-sieve size particles. (less than 37 microns). This is based on sedimentation process, using a hydrometer to secure the necessary data.

APPARATUS:

- a. Balance    b. Stirring apparatus    c. ASTM Hydrometer  
(152H used in this case), d. Sedimentation cylinder  
of capacity 1000 ml    e. Constant temperature bath
- f. Beaker of 250 ml capacity    g. Stop watch

CHEMICALS:

- a. 4 percent solution of sodium hexametaphosphate in  
distilled water    b. Distilled water

PROCEDURE:

1. Dispersion of Sample: 40 grams of sample were weighed in a 250 ml. beaker to which 100 ml. of 4 percent sodium hexametaphosphate solution were added. The slurry was well-stirred until it was thoroughly wet and then was allowed to soak for atleast 18 hours. At the end of soaking period, the sample was dispersed further, using stirring apparatus for about 10 minutes.

2. Hydrometer Reading: Immediately after dispersion the slurry was transferred to the glass sedimentation cylinder and distilled water was added to make up the volume to 1000 ml.

Using the palm of the hand over the open end of the cylinder, the cylinder was made upside down and back for a period of 1 minute to complete the agitation of the slurry. At the end of 1 minute, cylinder was set in a water bath and hydrometer readings at times 1/2, 1, 2, 5, 10, 15, 30, 60, 120, 240, 360, 480, 1440 minutes were noted down. Hydrometer was inserted about 20 to 25 seconds before a reading was due. As soon as reading was taken, the hydrometer was carefully removed and placed with a spinning motion in a graduated cylinder of clean distilled water.

#### CALCULATION PROCEDURE:

1. For calculation of weight percent of particles remaining in suspension at the level at which hydrometer was measuring the density of the suspension, the following formula was used:

$$W = \frac{Ra}{W_s} \times 100$$

where,

R = Hydrometer reading with composite corrections applied, cm\*

a = Correction factor for specific gravity of slurry particles\*\*

$W_s$  = Weight of the sample taken for test

2. For diameter of a particle corresponding to the percentage

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\*Important corrections are (1) Meniscus correction (2) Temperature correction and (3) Correction for dispersing agent.

\*\*Values of a, K and L has been taken from the Table No. I, II, and III respectively of 1958 Book of ASTM standards for testing soils Part 4. pp: 1119 to 1129.

by a given hydrometer readings, Stoke's Law has been used. For convenience in calculations, it may be written as follows:

$$D = K \sqrt{L/T}$$

where K = constant depending on the temperature of the suspension and the specific gravity of particles\*\*.

L = Distance from the surface of the suspension to the level at which the density of the suspensions was being measured, in cms (For 152H hydrometer and the sedimentation cylinder used, value of L varies according to the hydrometer readings). This distance is known as effective length.\*\*

T = Interval of time from beginning of sedimentation to the taking of readings, in minute. Grain size analysis for grounded glass has been shown in Fig. 9 by plotting the diameters of the particles on a log scale as the abscissa and the percentage smaller than the corresponding diameters to an arithmetic scale as the ordinate. Grain size analysis for glass, quartz and alumina has been tabulated in Tables 2A to 2C.

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\*\*Values of a, K and L has been taken from the Table No. I, II and III respectively of 1958 Book of ASTM standards for testing soils Part 4, pp:1119-1129.

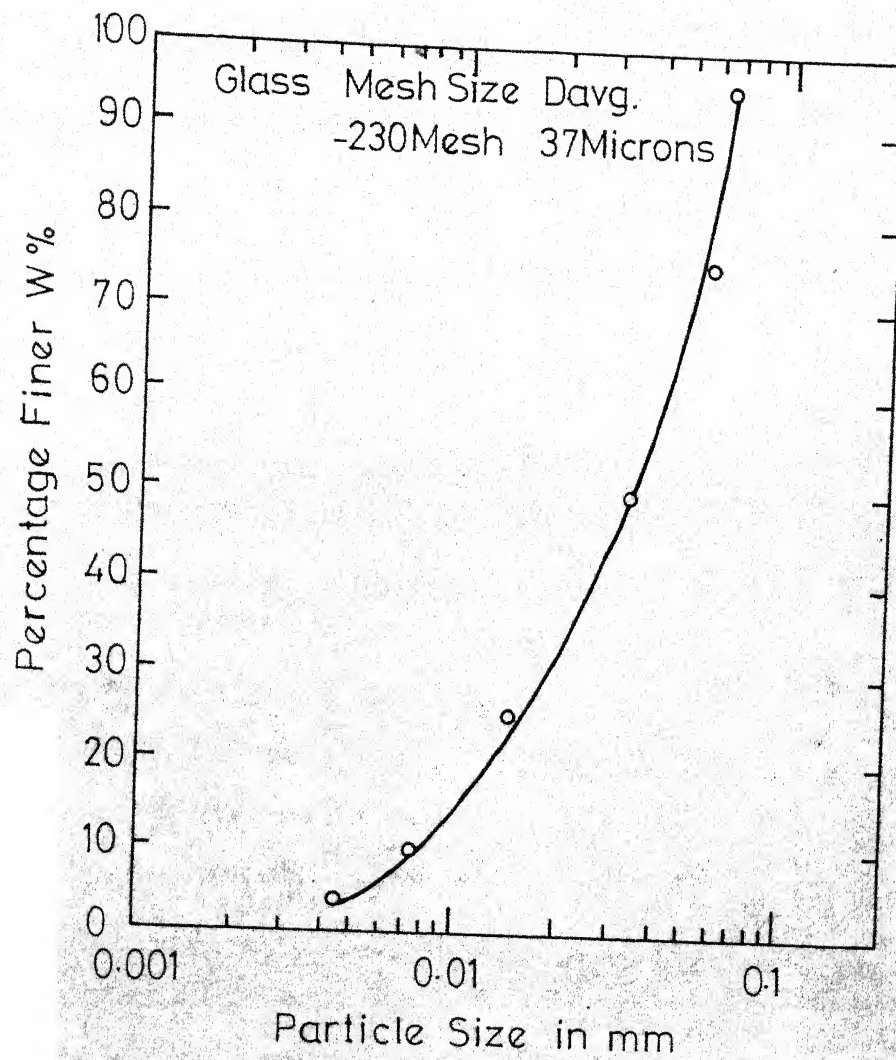


Fig. 9 - Grain Size Analysis.



APPENDIX BMETHOD FOR MEASUREMENT OF CONTACT ANGLE

Capillary rise technique<sup>(21)</sup> was employed to determine the contact angle of glass and quartz powders used in the experiment. This method consists in the unopposed rise of a liquid through a loosely packed column. The packed bed can be visualized as a number of interconnecting and tortuous capillaries of radius  $r$ . Washerman<sup>(22)</sup> has proposed following correlation for rise of a liquid through such a capillary.

$$\frac{l^2}{t} = \left( \frac{r}{K^2} \right) \frac{\gamma \cos \theta}{2\eta}$$

where

- $l$  = length of flow in time  $t$  seconds
- $\gamma$  = surface tension of the liquid
- $\eta$  = viscosity of the liquids
- $\theta$  = advancing contact angle
- $r/K^2$  = the tortuosity factor

Hence for a particular packing powders  $l^2/t$  will show a linear relationship, dependent only upon the nature of the liquid. If now a liquid be found for which  $\cos \theta = 1$  i.e. complete wetting occurs, a value of the tortuosity factor  $r/K^2$  can be obtained, enabling value of the adhesion tension  $\gamma \cos \theta$  to be obtained for other liquids of interest.

EXPERIMENTAL PROCEDURE:

A known weight of the dried powder was placed, and packed by tapping, in a glass tube, with an attached scale.

The glass tube selected was such that its internal diameter exceeded  $30d$ , where  $d$  is the diameter of power particles. This measure was aimed at minimizing the wall effect in wetting experiments. The lower end of the column was supported on a small plug of cotton wool covered with a disc of filter paper. The packed tube containing the powder was dipped into a dish of the liquid and the time at which the liquid commenced to wet the powder was observed. The height of the liquid in the column was subsequently observed at different intervals of time as indicated in the Figure 10 which shows an example of  $l^2/t$  plots for cleaned glass with benzene and water respectively.

On the assumption that Benzene completely wets the glass surface, contact angle for various solid particles with water has been tabulated below:

TABLE 4  
CONTACT ANGLE FOR COATED AND UNCOATED PARTICLES

Sl.No.	Solid	$D_{avg}$	Liquid	Contact angle
1.	Glass Sphere	74 Microns	Water	$5^\circ$
2.	Glass grounded	37 "	Water	$7^\circ$
3.	Quartz	63 "	Water	$9^\circ$
4.	Coated Glass	spherical as well as grounded	Water	More than $90^\circ$
5.	Coated Quartz	63 "	Water	" " $90^\circ$

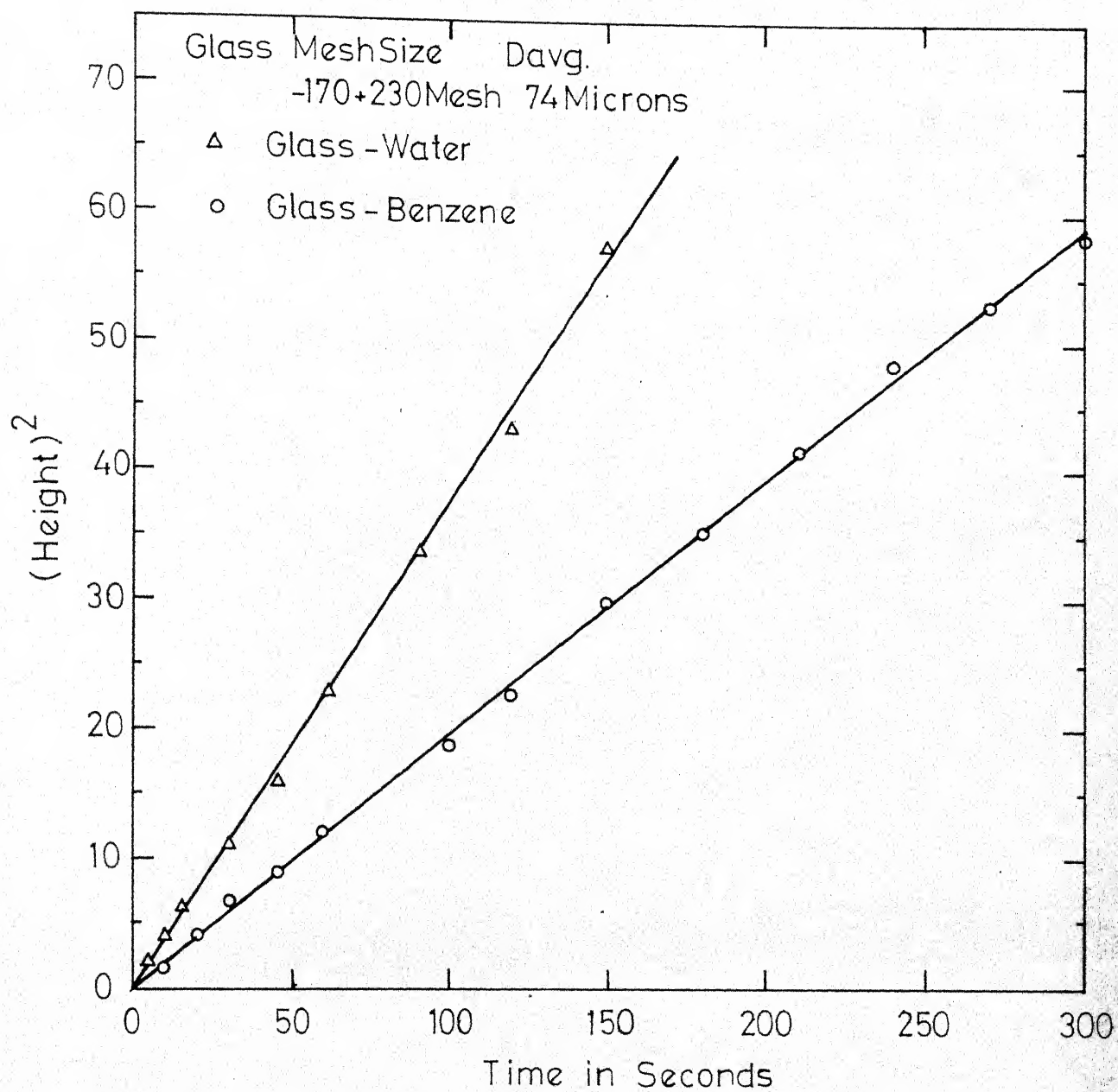


Fig.10 - Contact Measurement Plot.

APPENDIX CSAMPLE VISCOSITY DATA COLLECTION

Spindle Speed: 60 R.P.M.      Alumina Particles:  $D_{avg}$  14 Microns

Sl. No.	Volume Concentration*	Apparent Viscosity			
		1	2	3	Avg.
1.	6.00	2.05	1.95	1.95	1.95
2.	14.6	3.0	3.42	3.38	3.40
3.	24.2	9.30	9.25	9.25	9.25

\*Volume concentration is defined as volume of solids per unit volume of slurry